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**STRESS-STRAIN BEHAVIOR OF  
IRRADIATED POLYURETHANE ELASTOMERS**

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NUCLEAR AEROSPACE RESEARCH FACILITY

23 FEBRUARY 1962

**STRESS-STRAIN BEHAVIOR OF  
IRRADIATED POLYURETHANE ELASTOMERS**

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SECTION II, TASK III, ITEM 6  
OF FZM 2386

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## ABSTRACT

The applicability of the Martin-Roth-Stiehler stress-strain relationship to a number of reactor-irradiated (gamma-ray dose range:  $0 - 1.9 \times 10^{10}$  ergs/gram in carbon) polyurethane elastomers (DuPont Adiprene, Genthane, General Tire Polyurethane R, and Disogrin) has been demonstrated. For each level of dose and irradiation temperature, the stress-strain values showed remarkably good agreement with the behavior predicted by this relationship, thus permitting a realistic characterization of the stress-strain curves up to extension ratios of 3 by two parameters: the tangent modulus  $E_0$  and a coefficient  $K$ . While the available data did not justify conclusive inferences regarding the explicit nature of the functions  $E_0(D)$  and  $K(D)$ , it appears from this analysis that  $E_0$  varies exponentially with dose  $D$  and that  $K$  is a slowly varying function of dose.

## REPORT SUMMARY

The stress-strain behavior of a number of irradiated polyurethane elastomers was examined on the basis of an empirical stress-strain relationship developed by Martin, Roth, and Stiehler. While this relationship does not yield molecular information, it describes stress-strain behavior of vulcanizates much more adequately and over an extension range much greater than the theory of rubberlike elasticity. Applied to radiation-effects data, this relationship permits a realistic characterization of the stress-strain behavior by two parameters, the tangent modulus  $E_0$  and a coefficient  $K$ .

The data examined in this analysis are in remarkably good agreement with the behavior predicted by the Martin-Roth-Stiehler relationship, i.e., within each level of dose and irradiation temperature, the observed values follow closely the predicted straight-line course when the quantity  $\ln [f(D)\alpha^2/(\alpha - 1)]$  is plotted versus  $[\alpha^{-1}(\alpha^2 - 1)]$ , where  $f(D)$  and  $\alpha$  are the stress at dose  $D$  and the extension ratio, respectively.

Despite this internal consistency, no systematic correlation between dose and the two parameters  $E_0$  and  $K$  could be established. However, this analysis indicates that, in the majority of cases,  $\ln E_0$  increased with dose whenever  $K$  decreased and vice versa. For the specimens irradiated at 80°F, the predominant trend for  $\ln E_0$  was to increase with dose, whereas in the 260°F irradiations.

the predominant trend for  $\ln E_0$  was to decrease with dose. The parameter  $K$ , in general, appears to be a slowly varying function of dose, while the initial tangent modulus seems to vary exponentially with dose.

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## I. INTRODUCTION

The rheological behavior of elastomers is quite sensitively affected by high-energy radiation. Up until now, the theory of rubberlike elasticity has been used almost exclusively to evaluate radiation-induced changes in the stress-strain characteristics of these materials. This theory predicts that, in simple elongation or uniaxial compression, the force ( $f$ ) developed by a rubber sample per unit undeformed cross section is given by

$$f = G(\alpha - \alpha^{-2}) = (\rho RT/M_c)(\alpha - \alpha^{-2}), \quad (1)$$

where  $G$  = shear modulus,

$\alpha$  = extension ratio,

$\rho$  = density of the sample,

$R$  = universal gas constant,

$T$  = absolute temperature, and

$M_c$  = molecular weight between crosslinks.

Accordingly, the stress-strain behavior of elastomeric materials is described by this relationship in terms of a single elastic constant  $G$  which, in turn, is related to the number of network chains.

A widespread practice in radiation-effects studies has been to employ this equation for the assessment of radiation-induced changes in the molecular weight between crosslinks.

Experimental data for such an evaluation have been acquired principally on the basis of two investigational methods:

1. Stress-strain measurements after irradiation of the samples in an unstrained state;
2. Continuous stress relaxation tests in which the sample is held at constant elongation within the radiation field and the stress is measured as a function of dose.

In the first method essentially two types of measurements are conducted: (a) postirradiation percent moduli (stress at a specified percent elongation), and (b) intermittent stress relaxation tests. The two measurements are similar, except that in the first case, only one measurement is normally made per sample, whereas in the latter case, several measurements of stress are obtained from the same sample at specified intervals of time. Equilibrium conditions within the sample are not likely to be attained in the case of intermittent stress relaxation tests, while the chances therefor are much more favorable in the postirradiation measurement of percent modulus. Manifestly, continuous stress relaxation behavior involves a decay process whose rate is sensitively dependent on the scission reaction rate.

To the authors' knowledge, the applicability of Equation 1 to radiation-effects data has, to date, not been adequately scrutinized. Experimental evidence indicates that while this expression describes correctly the general form of stress-strain curves in the region of moderate extension ratios ( $\alpha \approx 5 - 1.5$ ), significant departures from it are encountered at very low strains and, more markedly, at high strains.

Furthermore, it must be kept in mind that the theory on which Equation 1 is based attributes structural equilibrium to the network - a condition which never really prevails, even in very short-term deformations. The implicit assumption in applying Equation 1 to radiation-induced stress relaxation has been to consider the system in a pseudo-equilibrium state. While this assumption may be justified to some extent in the case of intermittent measurements, it certainly cannot be held to be true in the case of continuous measurements.

Furthermore, in deducing molecular events from theory, investigators have, so far, not considered the influence of strain on reaction probabilities, although experimental evidence suggests that such strain dependence is quite real.

Apart from these considerations, there is the additional difficulty of verifying the molecular implications of the theory even within the range of its approximate validity. In all cases where molecular information is to be extracted from this relationship, a concurrent attempt should be made at providing an independent measure for the particular molecular events. Quantitative conclusions based solely on this relationship are not really justified.

Other theories have been developed which describe more satisfactorily the stress-strain behavior of elastomers in the regions of extension beyond the validity of Equation 1. Yet these theories have neither the simplicity nor the fundamental cogency of the theory of rubberlike elasticity.

However, an empirical equation developed in Reference 1 by Martin, Roth, and Stiehler (hereinafter referred to as the MRS relationship) has been found to describe, in a remarkably successful manner, the equilibrium stress-strain behavior of many elastomeric materials up to extension ratios of  $\alpha \approx 3$ .

According to the MRS relationship, the force developed by a rubber sample per unit undeformed cross section is given by

$$f = E_0(\alpha^{-1} - \alpha^{-2}) \exp \{ K(\alpha - \alpha^{-1}) \} \quad , \quad (2)$$

where  $E_0$  = tangent modulus at  $\alpha = 1$  and  
 $K$  = material constant.

This expression has recently been used by several investigators to determine the elastic modulus as a function of mechanical degradation and chemically induced scission and crosslinking processes. The MRS relationship was found to be applicable to a wide variety of vulcanizates. It was found to be less successful in describing the stress-strain behavior of vulcanizates containing carbon black and other fillers.

The idea suggested itself that, if the MRS relationship were applicable to irradiated elastomers, its use might contribute substantially towards a systematization of experimental data, since in this manner the stress-strain behavior can be characterized simply by two parameters,  $E_0$  and  $K$ . If, moreover, the dose dependence of these parameters could be determined, it would be possible to arrive at predictive relationships of more general utility which, in turn, might furnish valuable clues as to the underlying molecular processes.

## II. EXPERIMENTAL PROCEDURE AND DATA

### 2.1 Procedure

The MRS relationship (Equation 2) was rearranged in logarithmic form so that

$$\ln \left[ \frac{f(D)\alpha^2}{\alpha - 1} \right] = K(D) \left[ \alpha^{-1}(\alpha^2 - 1) \right] + \ln E_0(D), \quad (2a)$$

where D indicates the radiation dose dependence of the various parameters. Thus, by plotting the quantity  $\ln [f(D)\alpha^2/(\alpha - 1)]$  as a function of  $[\alpha^{-1}(\alpha^2 - 1)]$ , a straight line is obtained. The parameter  $K(D)$  is then equal to the slope of the straight line, and  $\ln E_0(D)$  is its intercept with the ordinate.

### 2.2 Data

The experimental data treated here were taken from recent work carried out at the Nuclear Aircraft Research Facility (Ref. 2\*). The test specimens consisted of various types of polyurethane elastomers (see Table I). The specimens were prepared according to the procedure prescribed for tensile specimens in the ASTM standards (ASTM-D 412-51T, Die C). Measurements of stress-strain values (percent modulus) and ultimate tensile strength were also carried out in accordance with this procedure

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\*This reference lists the arithmetic mean of the observed values, while the present investigation is based on a least-square analysis of the raw data that the author was kind enough to furnish.

TABLE I  
IRRADIATION DATA AND CALCULATED VALUES OF  $1/r$ ,  $E_0$  AND  $K$

NO.	SAMPLE DESCRIPTION	RUN	GAMMA DOSE [ergs gm(C)]	NEUTRON DOSE n cm <sup>2</sup>	IRRADIATION TEMPERATURE (°F)	$\ln E_0$	K
1.	DuPont Aliprene L, 100 parts; MOCA, 12 parts. Cure: 3 hours at 100°C	1 2 3 4	0 2 x 10 <sup>9</sup> 5.4 x 10 <sup>9</sup>	0 1.25 x 10 <sup>15</sup> 1.8 x 10 <sup>15</sup>	80 260 80 260	5.25 5.76 5.97 5.04	0.100 0.114 0.049 0.350
2.	DuPont L-167, 100 parts; MOCA, 18 parts. Cure: 1 hour at 100°C	1 2 3 4	0 2 x 10 <sup>9</sup> 5.1 x 10 <sup>9</sup>	0 1.25 x 10 <sup>15</sup> 1.8 x 10 <sup>15</sup>	80 260 80 260	5.47 5.79 5.60 5.79	0.528 0.382 0.500 0.280
3.	DuPont L-167, 100 parts; 1,4 butanediol, 5.8 parts; trimethylolpropane, 1 part. Cure: 1 hour at 110°C	1 2 3 4	0 2 x 10 <sup>9</sup> 5.1 x 10 <sup>9</sup>	0 1.25 x 10 <sup>15</sup> 1.8 x 10 <sup>15</sup>	80 260 80 260	3.14 3.44 3.76 3.27	0.472 0.413 0.310 0.263
4.	Genthane S-1	1 2 3 4 5	0 1.3 x 10 <sup>10</sup> 1.5 x 10 <sup>10</sup> 7 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 6.7 x 10 <sup>15</sup> 4.5 x 10 <sup>16</sup>	80 260 -65 80 80	3.33 2.88 2.54 3.39 1.39	0.820 1.00 1.02 0.836 1.91
5.	Genthane S-2	1 2 3 4 5	0 1.3 x 10 <sup>10</sup> 1.5 x 10 <sup>10</sup> 1.9 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 6.7 x 10 <sup>15</sup> 8.5 x 10 <sup>15</sup>	80 260 -65 80 260	2.81 2.28 2.89 2.44 1.96	1.015 1.255 1.02 1.265 1.68
6.	General Tire Polyurethane Type R	11 2 3 4 5	0 1.3 x 10 <sup>10</sup> 1.5 x 10 <sup>10</sup> 1.9 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 6.7 x 10 <sup>15</sup> 8.5 x 10 <sup>15</sup>	80 260 -65 80 260	3.19 2.56 2.50 6.32 3.11	0.838 1.075 0.919 -0.467 1.28
7.	Disogrin 1, DSA6575	1 2 3 4 5	0 1.3 x 10 <sup>10</sup> 1.5 x 10 <sup>10</sup> 1.9 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 6.7 x 10 <sup>15</sup> 8.5 x 10 <sup>15</sup>	80 260 -65 80 260	4.46 4.26 5.0 4.34 3.99	0.523 0.605 0.326 0.955 0.605
8.	Disogrin 1, DSA9250	1 2 3 4	0 1.3 x 10 <sup>10</sup> 1.9 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 8.5 x 10 <sup>15</sup>	80 260 -65 260	5.74 5.73 5.75 5.68	0.333 0.325 0.263 0.362
9.	Disogrin 2, DSA9848	1 2 3 4 5	0 1.3 x 10 <sup>10</sup> 1.5 x 10 <sup>10</sup> 1.9 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 6.7 x 10 <sup>15</sup> 8.5 x 10 <sup>15</sup>	80 260 -65 80 260	5.41 5.41 6.03 5.8 6.0	0.389 0.389 0.060 0.208 0.016
10.	Disogrin 2, DSA7560	1 2 3 4	0 1.3 x 10 <sup>10</sup> 1.5 x 10 <sup>10</sup>	0 5.3 x 10 <sup>15</sup> 6.7 x 10 <sup>15</sup>	80 260 -65 80	4.26 3.97 4.60 4.64	0.486 0.575 0.394 0.570

for the controls as well as irradiated specimens. The latter were irradiated in the unstressed state in the Ground Test Reactor (GTR). The irradiation was conducted at three different temperatures:  $-65^{\circ}\text{F}$ ,  $80^{\circ}\text{F}$ , and  $260^{\circ}\text{F}$ . The control specimens were subjected to the same temperature regimen except that no controls were run at  $-65^{\circ}\text{F}$ . The specified temperature was maintained from one hour before irradiation until one hour after termination of irradiation. Thereafter, all specimens were stored at room temperature for approximately 30 days prior to the stress-strain measurements. These measurements were performed at approximately  $80^{\circ}\text{F}$ .

Specimens were irradiated at various dose levels in the range of from  $2 \times 10^9$  to  $1.9 \times 10^{10}$  ergs/gram in carbon ( $\gamma$ -ray dose) plus the dose imparted by the associated neutron flux (see Table I). A set of five tensile specimens were irradiated for each combination of dose, irradiation temperature, and extension ratio.

Dosimetric measurements of the dose absorbed from the  $\gamma$ -ray component of the reactor radiation were conducted with nitrous-oxide and tetrachloroethylene dosimeters, while the associated neutron fluxes were measured by means of aluminum foils and sulfur pellets. Whenever this type of gamma-ray dosimetry was not feasible on account of irradiation temperature, gamma-ray doses were obtained from neutron doses by the use of known neutron-to-gamma ratios.

Neutron fluxes were modified to include all neutrons of energy  $E > 0.33$  Mev by the use of appropriate factors derived from the GTR neutron spectrum (Refs. 3, 4).



### III. DISCUSSION OF RESULTS

In Figures 1 through 10, the experimental data on the various polyurethane elastomers, listed in Table I, are plotted according to the procedure outlined above, i.e., the quantity  $\ln [f(D)\alpha^2/(\alpha - 1)]$  is represented as a function of  $[\alpha^{-1}(\alpha^2 - 1)]$ . The resultant straight lines were fitted to their appertaining experimental points by a least-square analysis.

On the whole, the data are in surprisingly good agreement with the behavior predicted by the MRS relationship despite the fact that for the first 4 polyurethane compounds, extension ratios beyond 3 (i.e., beyond the accepted upper limit of validity of the MRS relationship) were included in the least-square analysis. The values of ultimate tensile strength and elongation were not considered in this investigation, because the measurement of tensile strength involves rheological processes to a significantly greater extent than in simple tensile quasi-equilibrium stress-strain measurements. Moreover, the numerical values, in most instances, exceeded the range of applicability of the MRS equation.

No systematic correlation between dose and the two parameters  $E_0$  and  $K$  could be established. Although there is significant internal consistency in stress-strain behavior for each level of dose and irradiation temperature, with the exception of one case (Figure 6 dose level  $1.5 \times 10^{10}$  ergs/gram in carbon, 80°F). In the majority of cases, it was noted that whenever  $\ln E_0$  increased with dose,  $K$  decreased and vice versa. For the specimens irradiated

at 80°F, the predominant trend for  $\ln E_0$  was to increase with dose, whereas in the case of the 260°F irradiations, the predominant trend for  $\ln E_0$  was to decrease with dose. It appears that, in general, parameter K is a slowly varying function of dose, while the initial tangent modulus varies exponentially with dose.

Since the experimental data used here were not designed specifically with this analysis in mind, certain inadequacies and errors are encountered that can be substantially eliminated in future experiments. However, since there is a definite internal consistency in each set of tests, the applicability of the MRS relationships to the irradiated polyurethane vulcanizates examined can be held to have been conclusively demonstrated. Two major sources of error are conjectured to be, at least partially, responsible for the apparent lack of systematicity in the behavior of the parameters  $E_0$  and K as a function of dose: (a) dosimetric inaccuracies and (b) irregularities in the temperature history of the specimens. For example, very little could be ascertained as to the exact course of the temperature treatment, particularly during the period between withdrawal from the reactor and time of measurement. Finally, other uncontrolled environmental factors, such as ozone generation in the reactor, may have adversely influenced the results.

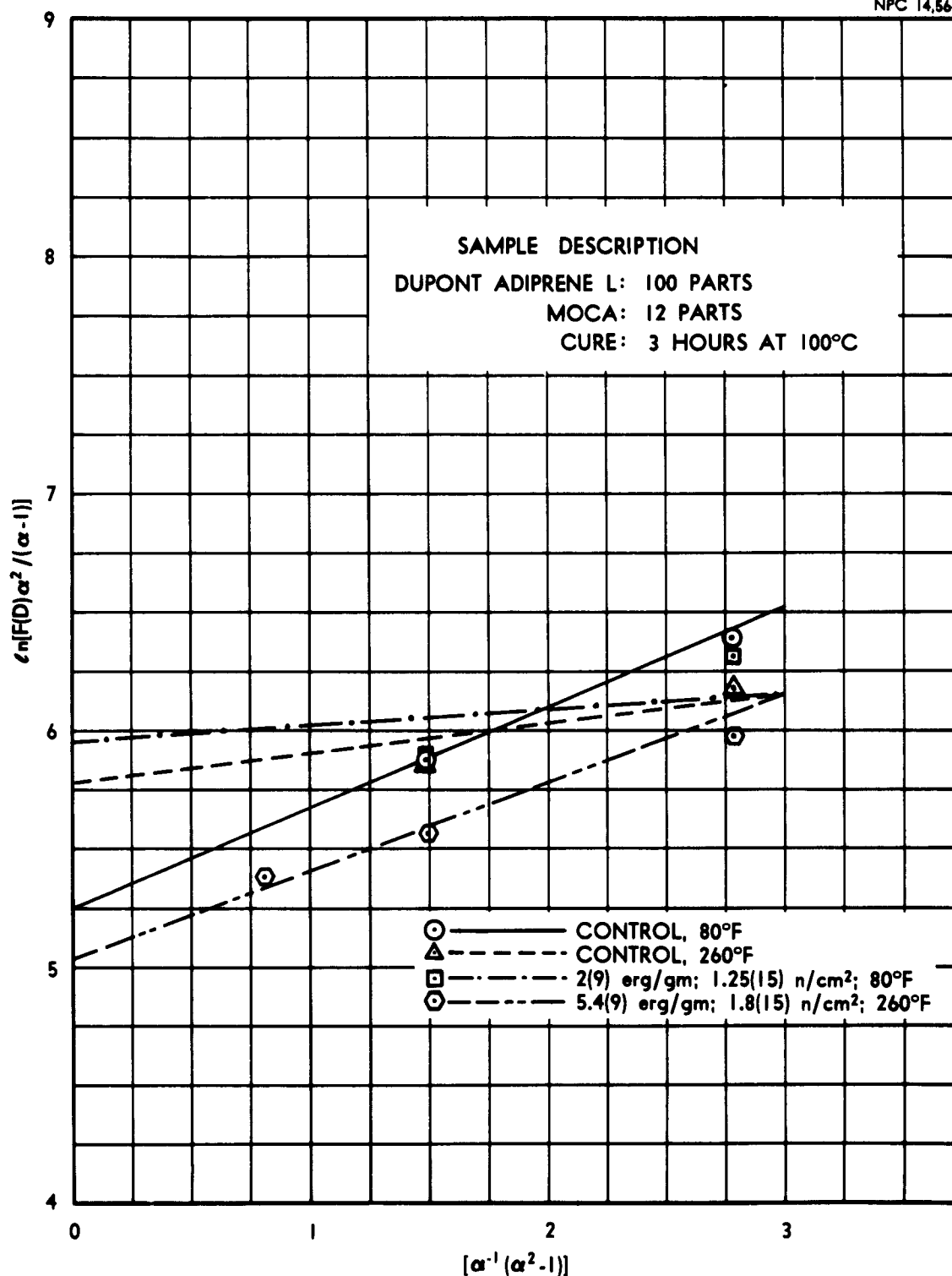
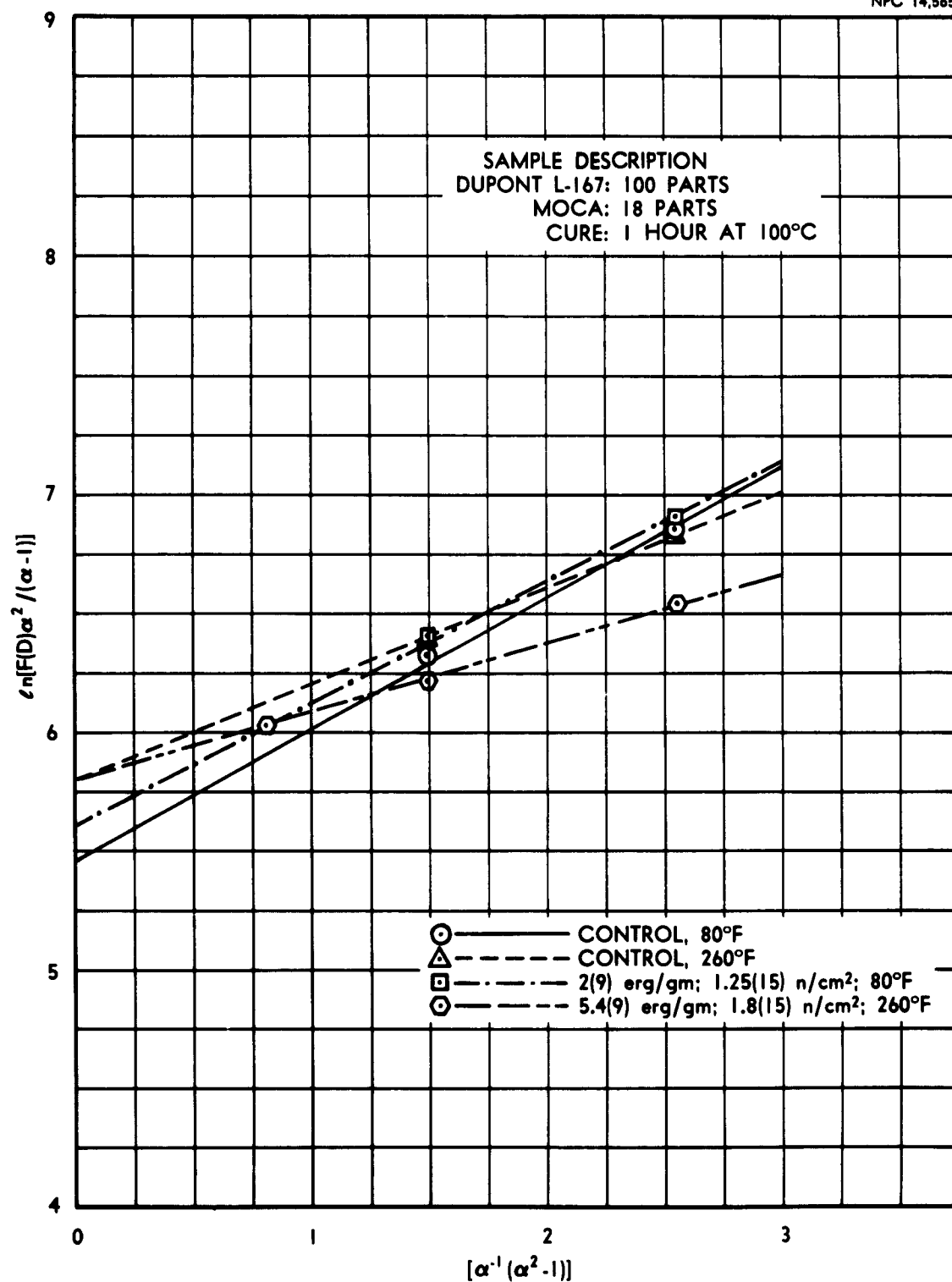


FIGURE 1. STRESS-STRAIN BEHAVIOR OF IRRADIATED  
 POLYURETHANE ELASTOMERS: SAMPLE 1



**FIGURE 2. STRESS-STRAIN BEHAVIOR OF IRRADIATED  
POLYURETHANE ELASTOMERS: SAMPLE 2**

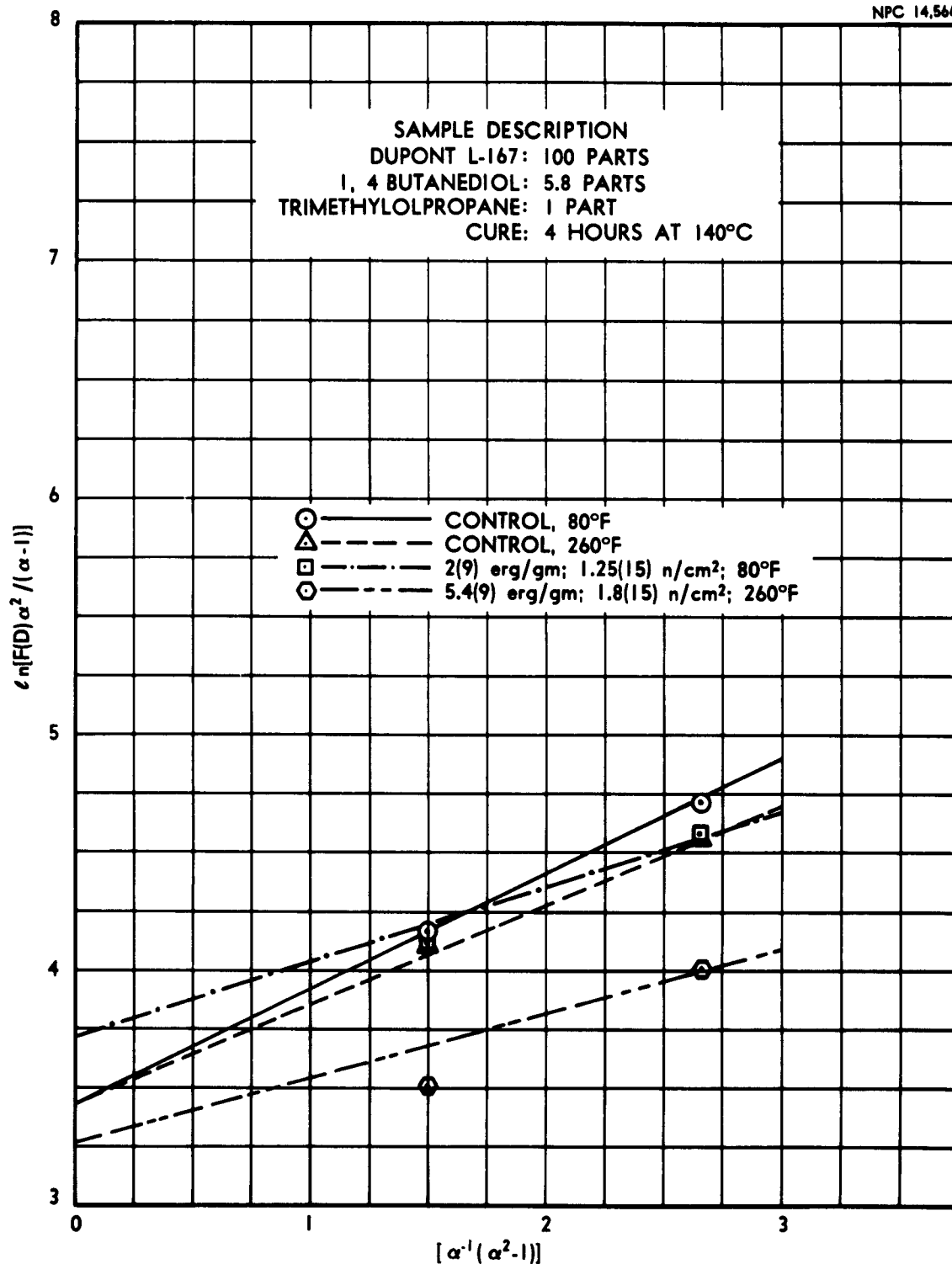


FIGURE 3. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS; SAMPLE 3

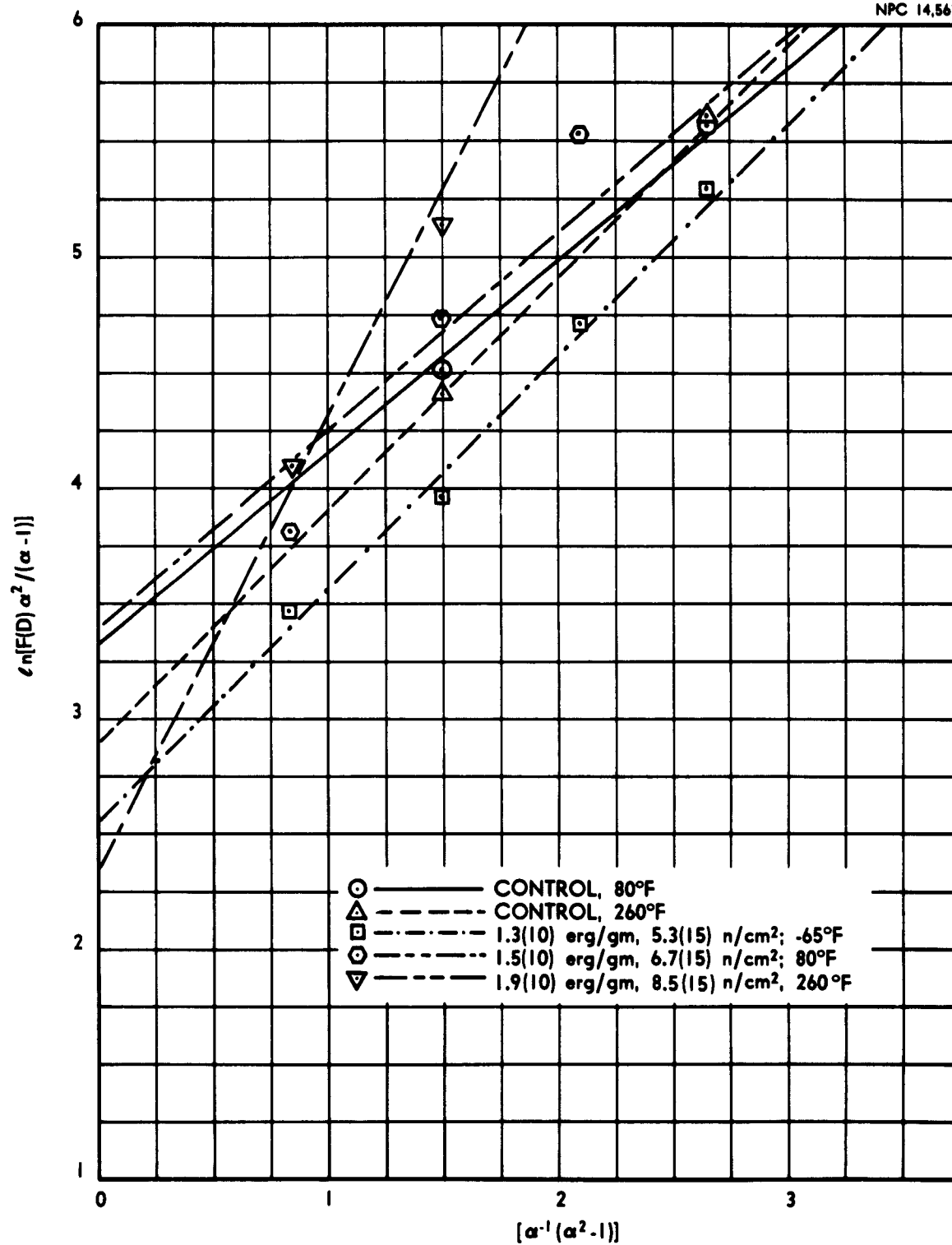
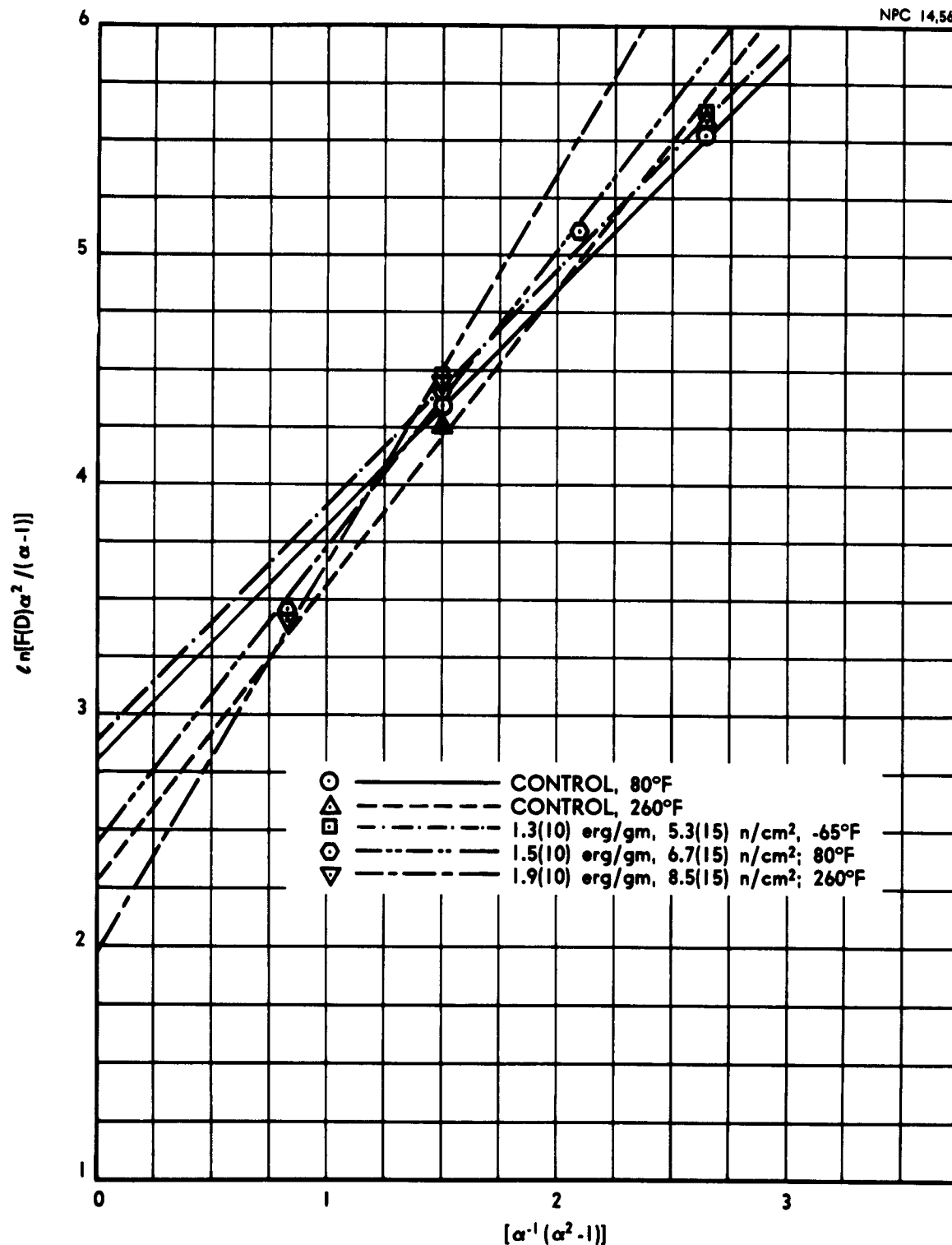


FIGURE 4. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 4 (GENTHANE S-1)



**FIGURE 5. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 5 (GENTHANE S-2)**

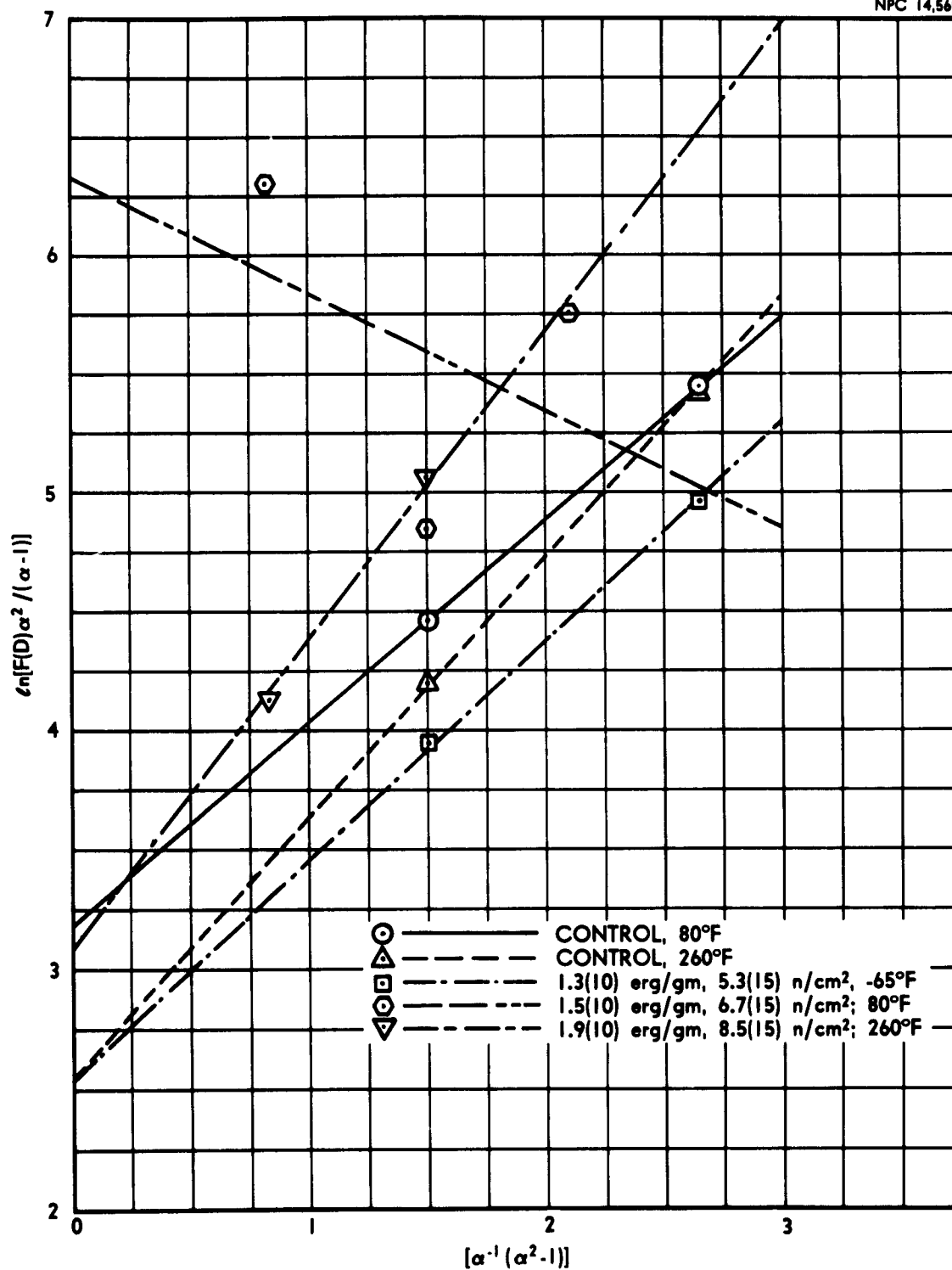
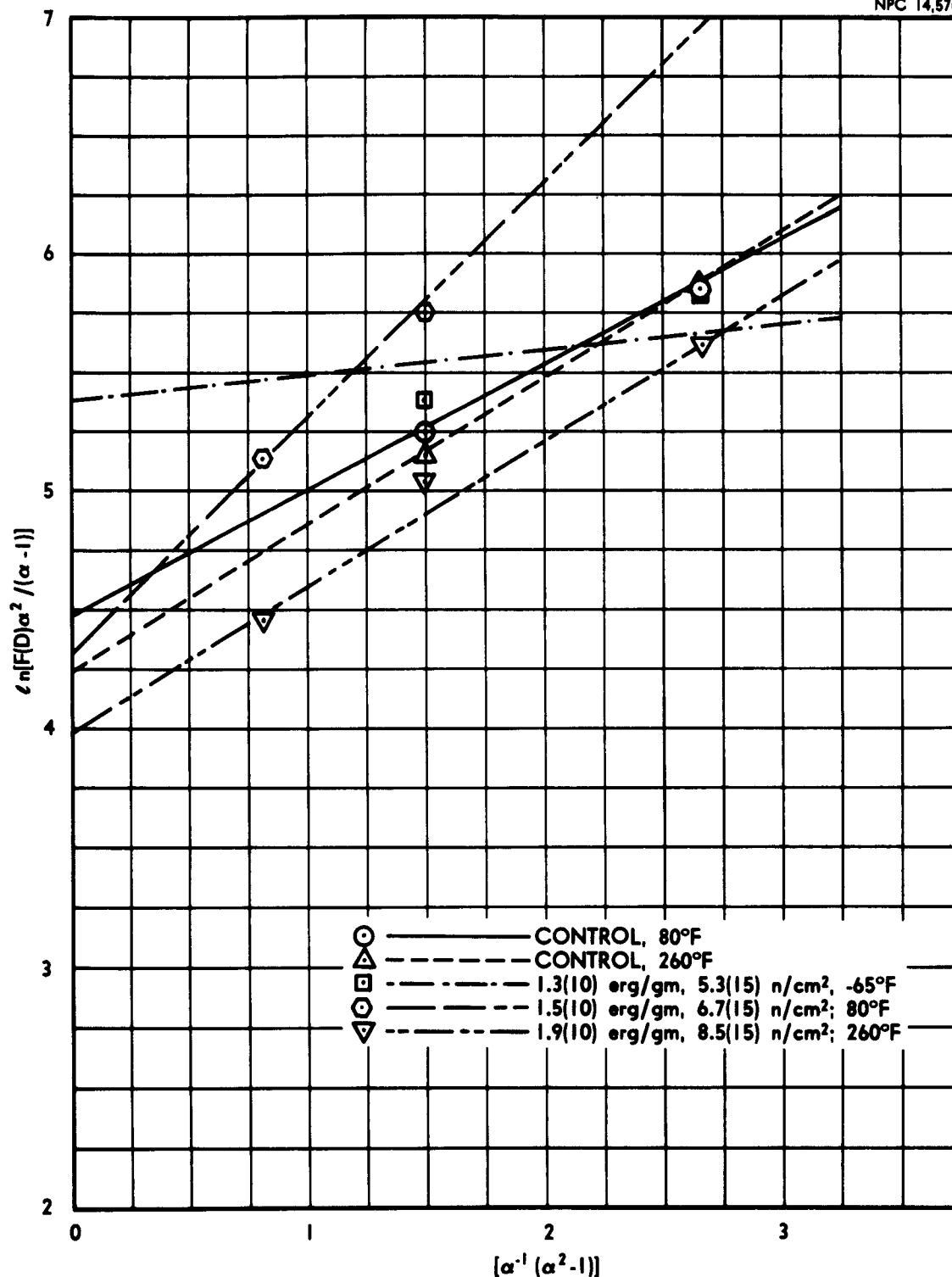


FIGURE 6. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 6 (GENERAL TIRE POLYURETHANE TYPE R)





**FIGURE 7. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 7 (DISOGRIN 1 DSA 6865)**

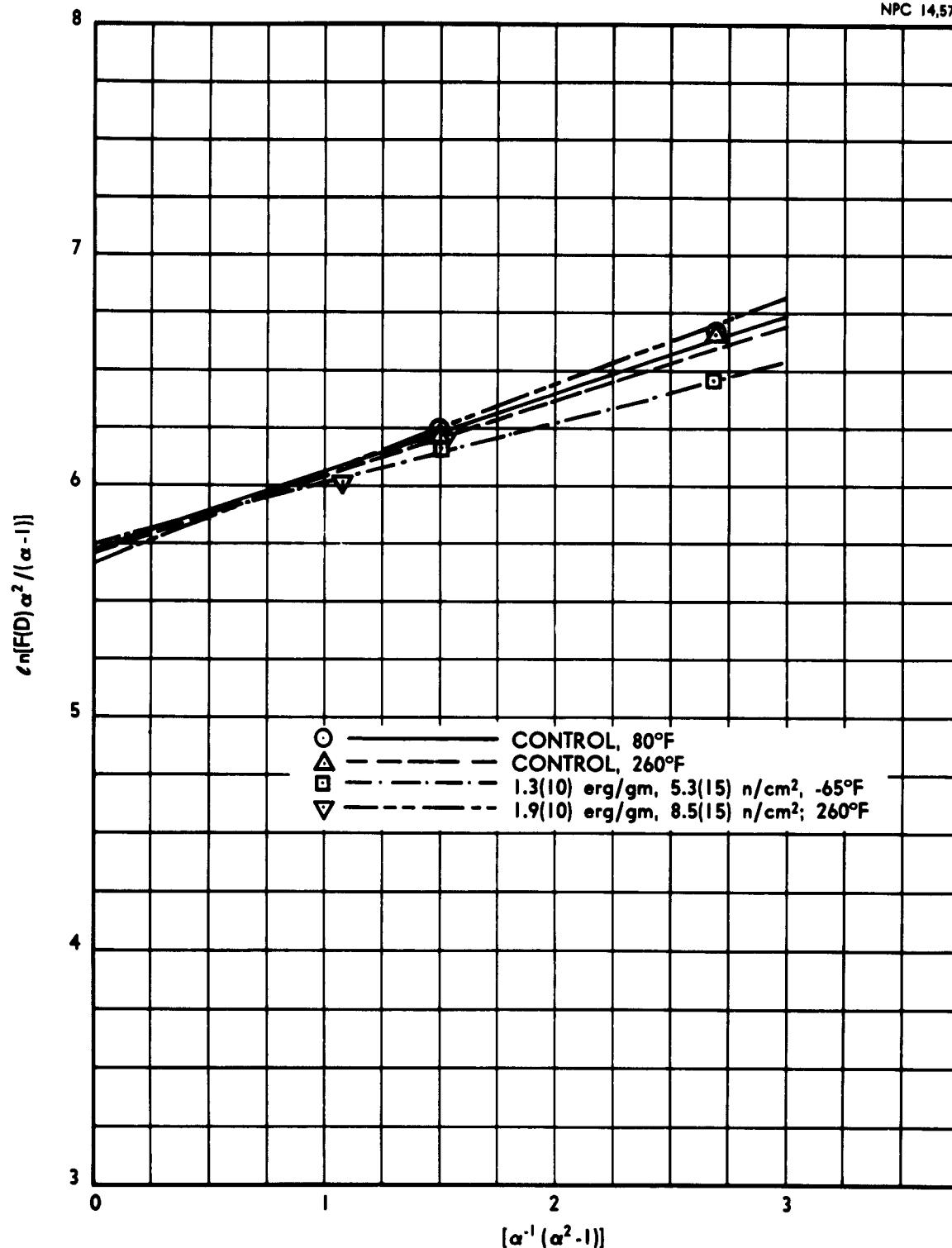


FIGURE 8. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 8 (DISOGRIN 1, DSA 9250)

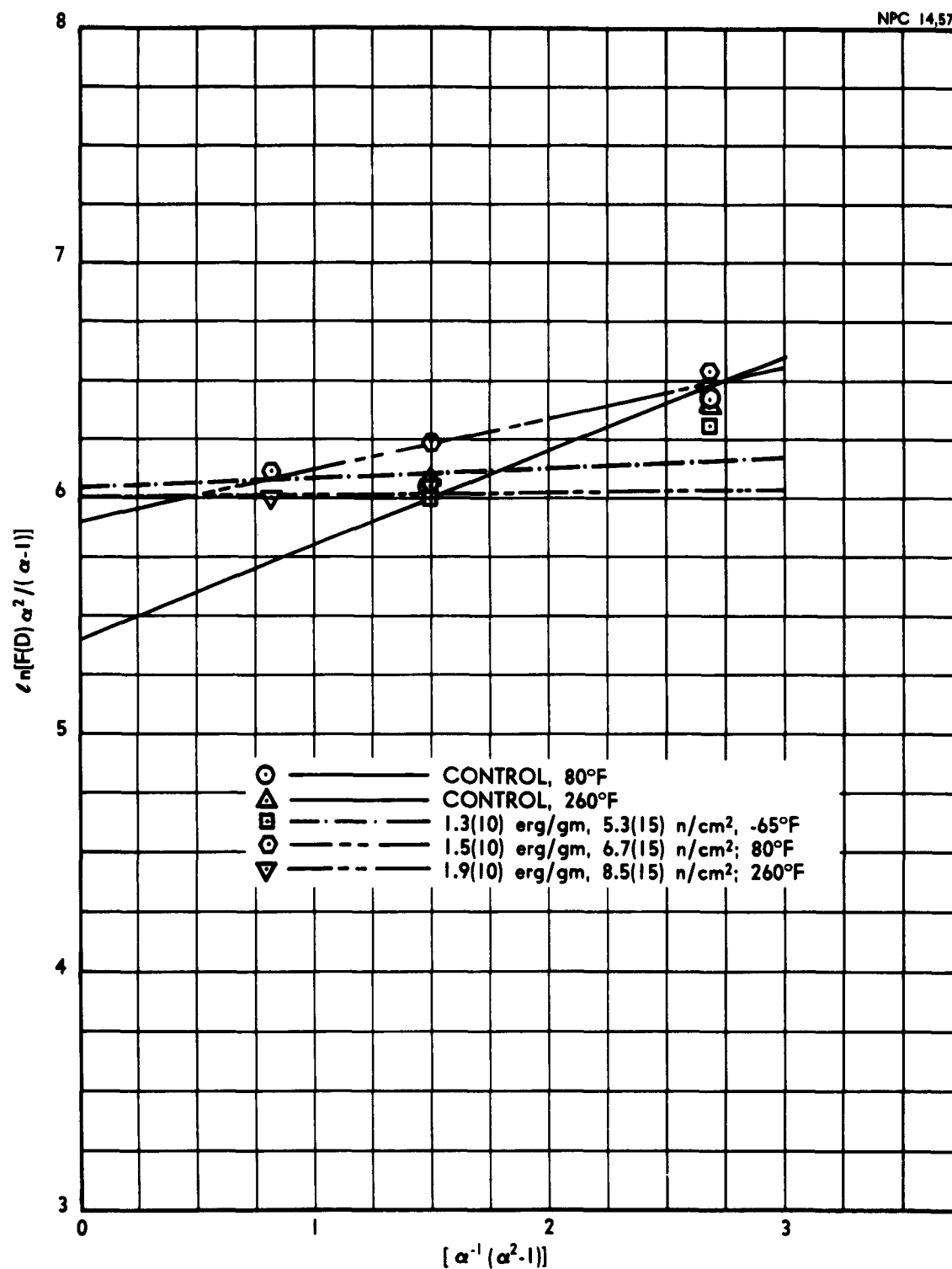


FIGURE 9. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 9 (DISOGRIN 2, DSA 9840)

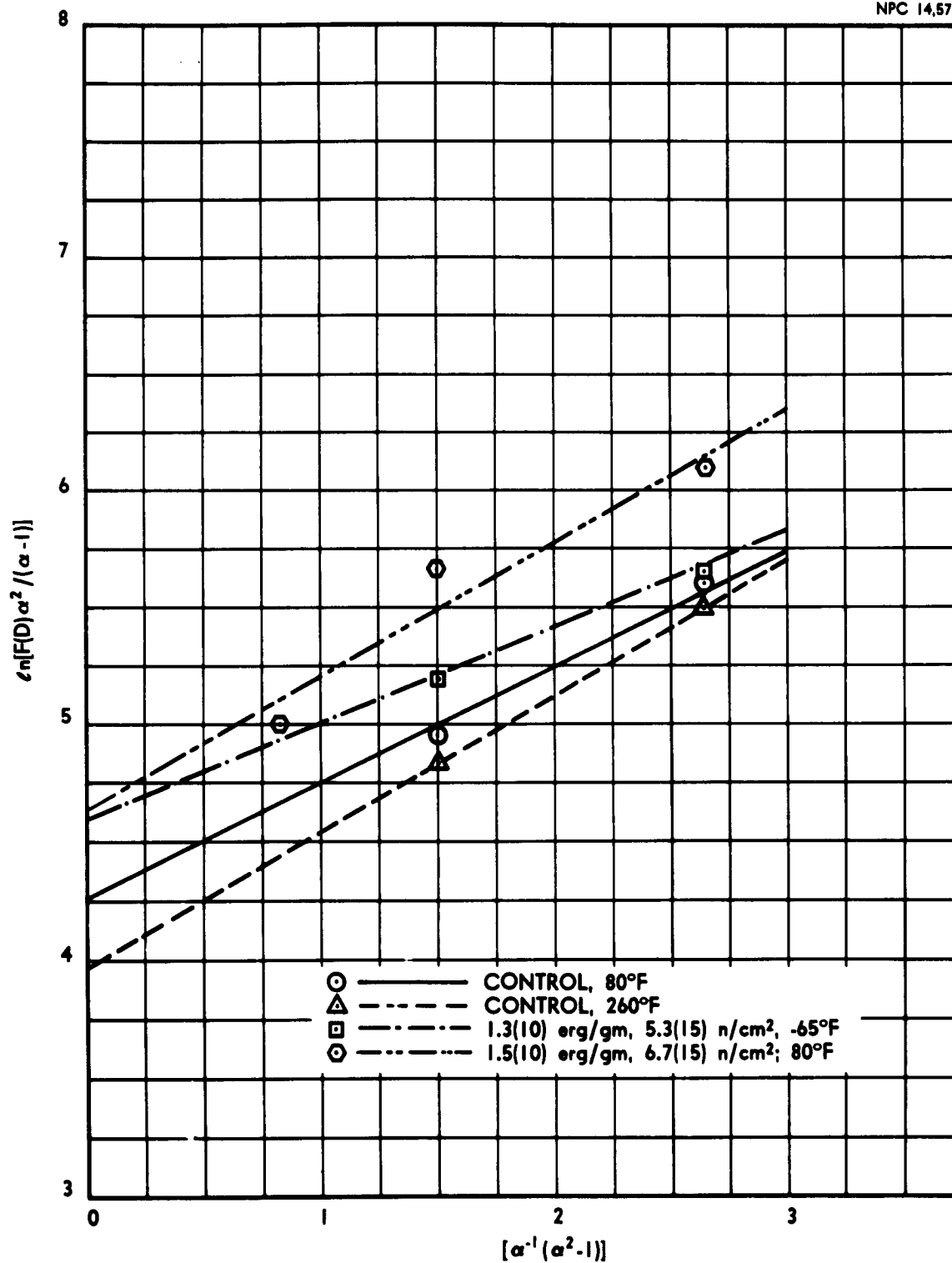


FIGURE 10. STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS: SAMPLE 10 (DISOGRIN 2, DSA 7560)

#### IV. CONCLUSIONS

The stress-strain behavior of irradiated polyurethane vulcanizates examined in this investigation showed very good agreement with the behavior predicted by the Martin-Roth-Stiehler relationship. With one exception, this agreement could be demonstrated for each level of dose and irradiation temperature. Although this relationship does not yield molecular information, it describes stress-strain behavior of elastomers much more adequately and over an extension ratio much greater than the theory of rubberlike elasticity.

Applied to radiation effects data, the MRS relationship permits a realistic characterization of the stress-strain behavior by two parameters,  $E_0$  and  $K$ , up to an extension ratio of 3. The available data did not justify conclusive inferences regarding the explicit nature of the functions  $E_0(D)$  and  $K(D)$ . It appears from the results of this examination that, in general, parameter  $K$  is a slowly varying function of dose, while the initial tangent modulus probably varies exponentially with dose.

It is suggested that this approach is of great utility in systematizing the description of stress-strain behavior of irradiated vulcanizates. Future stress-strain tests can, with little modification, be conducted in a manner which will yield more explicit information on the effects of dose and irradiation temperature on  $E_0$  and  $K$ . In order to achieve this objective, the following specific recommendations are made:

1. Stress-strain tests should be carried out at least at four different extension ratios (preferably at 1.5, 2, 2.5, and 3).
2. The speed of testing should be so adjusted as to minimize both the contribution from relaxation processes and from adiabatic heating. In each case, tension set should be measured.
3. Samples should be wrapped in aluminum foil to preclude ozonolytic action.
4. The temperature history of the sample must be more accurately recorded. Initially, both irradiation and testing should be done at room temperature to establish beyond doubt the validity of this relationship as a function of dose.
5. Irradiations should preferably be carried out in a pure gamma-ray field.
6. Testing according to ASTM procedure D412-51T is entirely satisfactory for this purpose if the above considerations are kept in mind.

In conclusion, it may be remarked that once an explicit functional correlation between dose and the parameters  $E_0$  and  $K$  is established, the MRS relationship may prove very helpful in elucidating the strain dependence of radiation effects.

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<p>Nuclear Aerospace Research Facility, General Dynamics/Port Worth, Fort Worth, Texas.</p> <p>STRESS-STRAIN BEHAVIOR OF IRRADIATED POLYURETHANE ELASTOMERS, by E. G. Fritz and P. M. Johnson. February 1962. 29p. incl. illus. table, 4 refs. (NARP-62-2T; MR-N-293) Unclassified report (Contract AF 33(657)-7201)</p> <p>The applicability of the Martin-Roth-Stiehler stress-strain relationship to a number of reactor-irradiated (gamma-ray dose range: 0 - <math>1.9 \times 10^{10}</math> ergs/gram in carbon) polyurethane elastomers (DuPont Adiprene, Genthane, General Tire Polyurethane R, and Disogrin) has been demonstrated. For each level of dose and irradiation temperature, the stress-strain values showed remarkably good agreement with the behavior predicted by this relationship, thus permitting a realistic characterization of the stress-strain curves up to extension ratios of 3 by two parameters: the tangent modulus <math>E_0</math> and a coefficient K. While the available data did</p>	<p>UNCLASSIFIED</p> <p>1. Radiation damage - Analysis</p> <p>2. Polymers - Effects of radiation</p> <p>3. Elastomers - Mechanical properties</p> <p>4. Effects of radiation - Tensile properties</p> <p>I. Johnson, P. M. II. Fritz, E. G. III. Aeronautical Systems Division, Air Force Systems Command IV. Contract AF 33(657)-7201</p> <p>UNCLASSIFIED</p>	<p>UNCLASSIFIED</p> <p>1. Radiation damage - Analysis</p> <p>2. Polymers - Effects of radiation</p> <p>3. Elastomers - Mechanical properties</p> <p>4. Effects of radiation - Tensile properties</p> <p>I. Johnson, P. M. II. Fritz, E. G. III. Aeronautical Systems Division, Air Force Systems Command IV. Contract AF 33(657)-7201</p> <p>UNCLASSIFIED</p>	<p>UNCLASSIFIED</p> <p>1. Radiation damage - Analysis</p> <p>2. Polymers - Effects of radiation</p> <p>3. Elastomers - Mechanical properties</p> <p>4. Effects of radiation - Tensile properties</p> <p>I. Johnson, P. M. II. Fritz, E. G. III. Aeronautical Systems Division, Air Force Systems Command IV. Contract AF 33(657)-7201</p> <p>UNCLASSIFIED</p>
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